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Fire Science & Technology

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Carbon Fiber Epoxy Hazards from Fire Environment Testing

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Fire Science and Technology Department

SAND2014-????C

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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Background & Motivation

- Carbon fiber epoxy composite fire testing has been ongoing at Sandia to discover the effect of these materials on a fire environment.
- Thermal behavior was always the primary motivation, but secondary objectives were also pursued
- The literature suggests peculiar concerns relative to the health hazards from fires involving these materials
  - Post clean-up complaints by response personnel
  - Lung and skin irritation from residual fiber and smoke
- Several hazard types are known to be present
  - Toxic fumes (CO, CHN, PAHs)
  - Soot
  - Fiber particulates in the respirable range

Objectives

- Secondary objectives relating to carbon fiber composite fire included the quantification of various potential health hazards
- Two test series provided thermal data as the primary objective, but the secondary data provide some useful information about such fires
- This presentation exhibits the findings from the secondary data

Outline

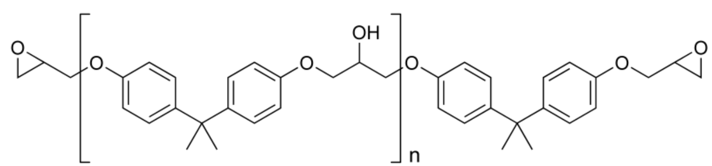
- Introduce and present soot data from enclosure fire tests
- Introduce and present particulate and fiber data from mock fuselage fire tests
- Discussion of the implications of the data

Background Composition Information

- Around ~35% epoxy, ~65% carbon fiber
- Fabric (woven) or uni-tape sheets, usually multiple layers thick
- Possibly sandwich material with high void fraction material between two composite sheets
- Pressed and cured in an autoclave, or similar
- Fibers around 5 μm diameter, 95% carbon

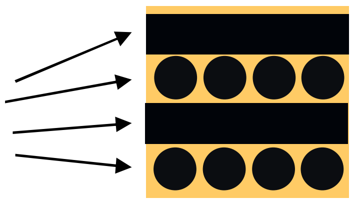
Epoxy (DEGBA) and TETA hardener (From wikipedia):

$[C_{18}H_{20}O_3]_n$



A four layer cross-section illustration:

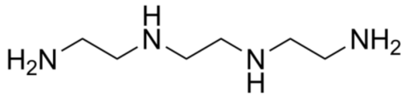
Fibers in varying orientation



Key:

Carbon Fibers

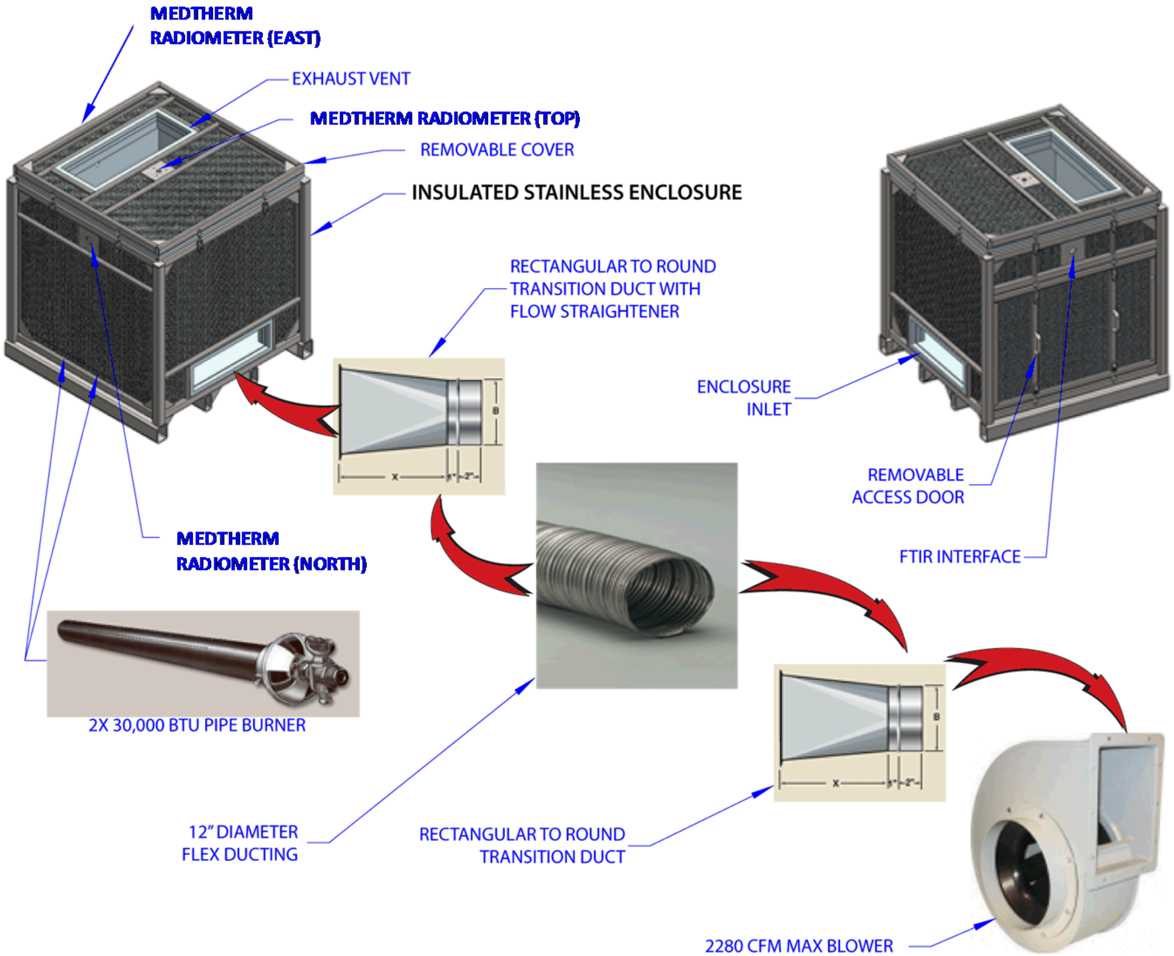
Epoxy Resin



$C_6H_{18}N_4$

Test Enclosure

- 91.0 cm aspirated internal cube designed to create an idealized semi-adiabatic environment



<u>Instrumentation</u>

- FTIR
- RGA/Mass Spec.
- Radiometers
- Calorimeter
- Thermocouples
- Pitot Velocity Probe
- Video

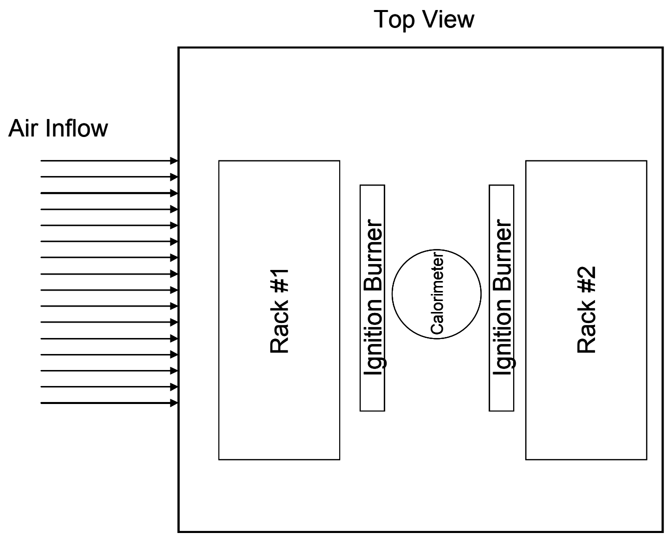
Test Matrix

- Tests 1 and 2 were scoping tests performed with wood
- Tests 3 and 4 were preliminary tests with scrap composites
- Tests 5, 6, and 7 were more heavily instrumented, involved a more regular arrangement of the composite material
  - Tests 5 and 6 included soot sampling
  - Tests 5 and 7 included FTIR gas species sampling

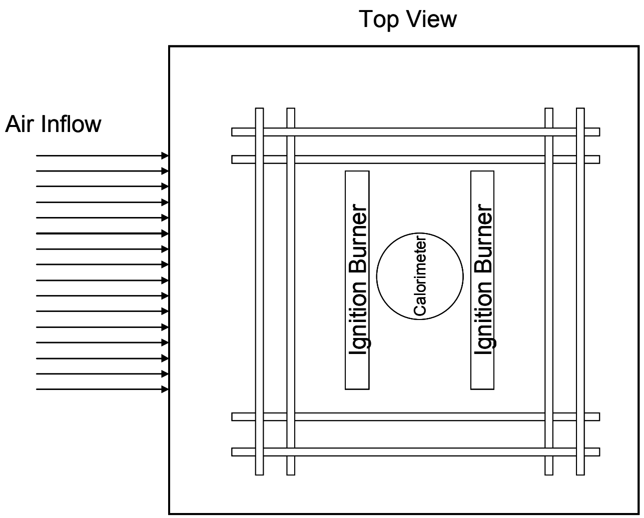
Parameter	Test 5	Test 6
Material Description	Body Armor	Strips
Manufacturer	Hercules	Hexcel
Epoxy	3501-6 resin	3501-6 resin
Fibers	AW370 woven carbon fibers	Woven carbon fibers
Mass	36.6 kg	39.3 kg
Est. SA/Vol ratio	2.0 cm^{-1}	9.2 cm^{-1}
Arrangement	Two racks	Crib

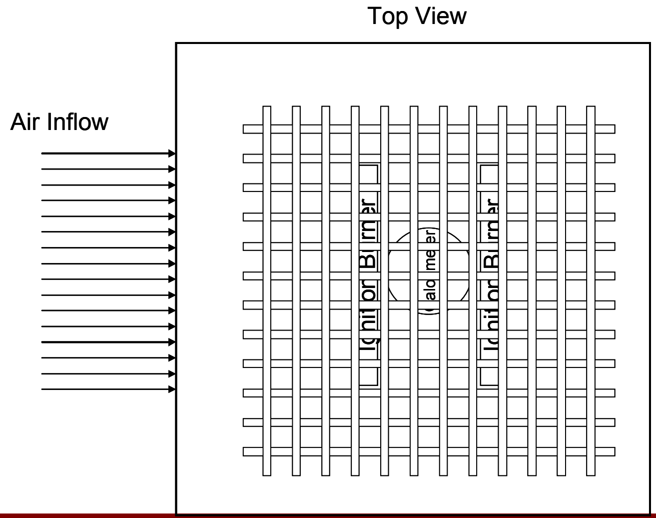
Test 5 & 6 Layout

Test 5



Test 6





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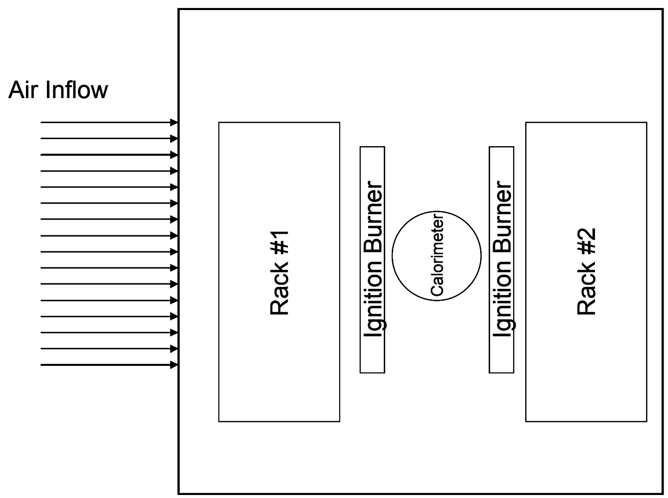
Test 5 & 6 Layout

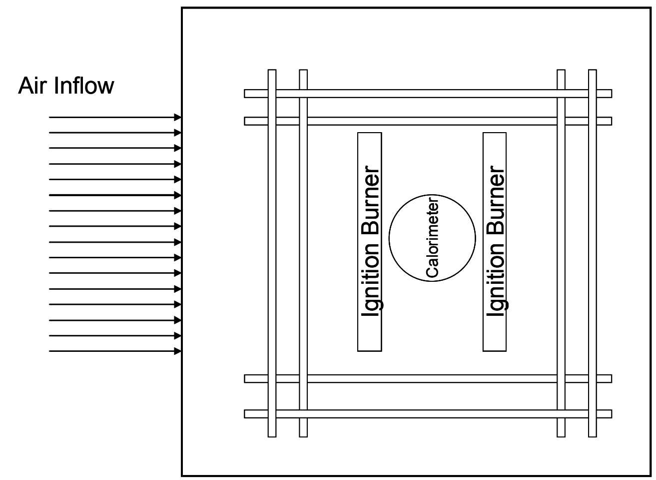
Test 5

Top View



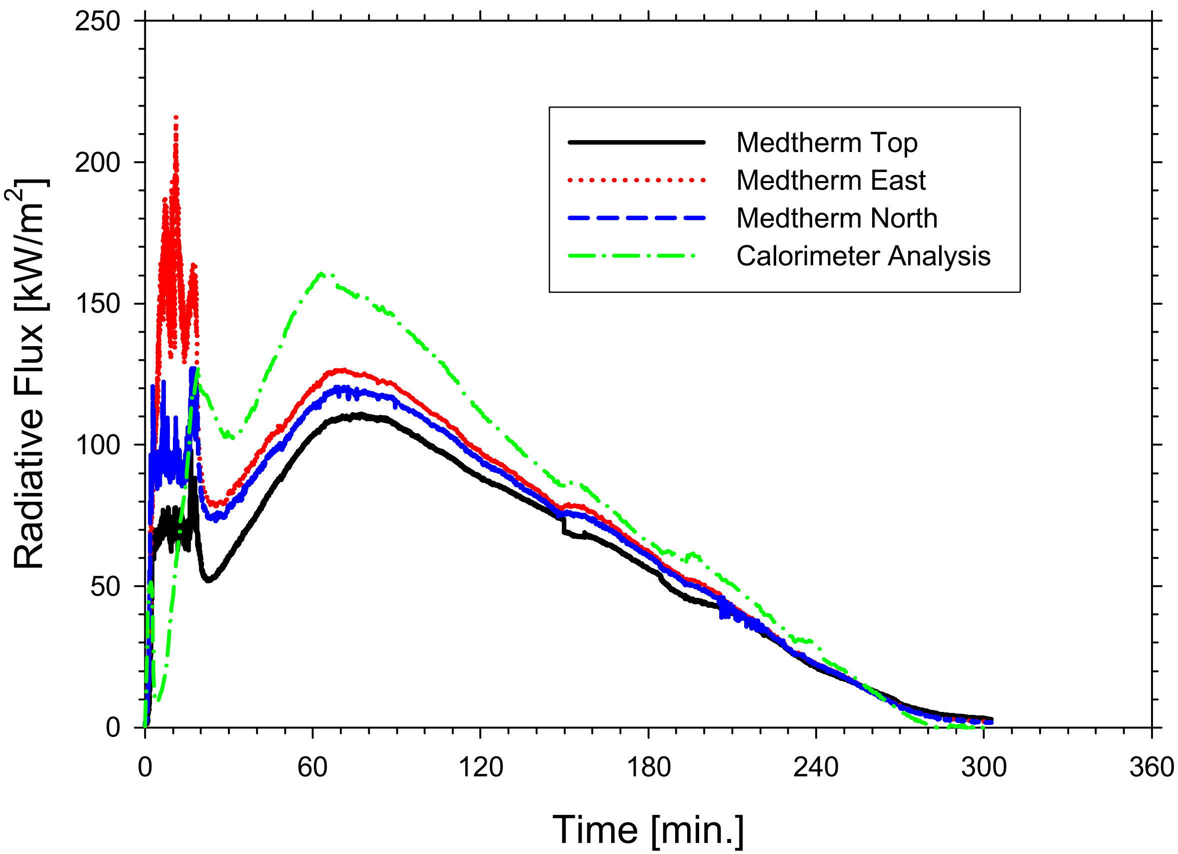
Test 6

Top View



Top View

Typical (Test 6; Strips) Thermal Results



Calorimeter shows lower flux during flaming, higher during glowing combustion times
Fluxes as high as 150 kW/m^2 are found during glowing combustion in the bed

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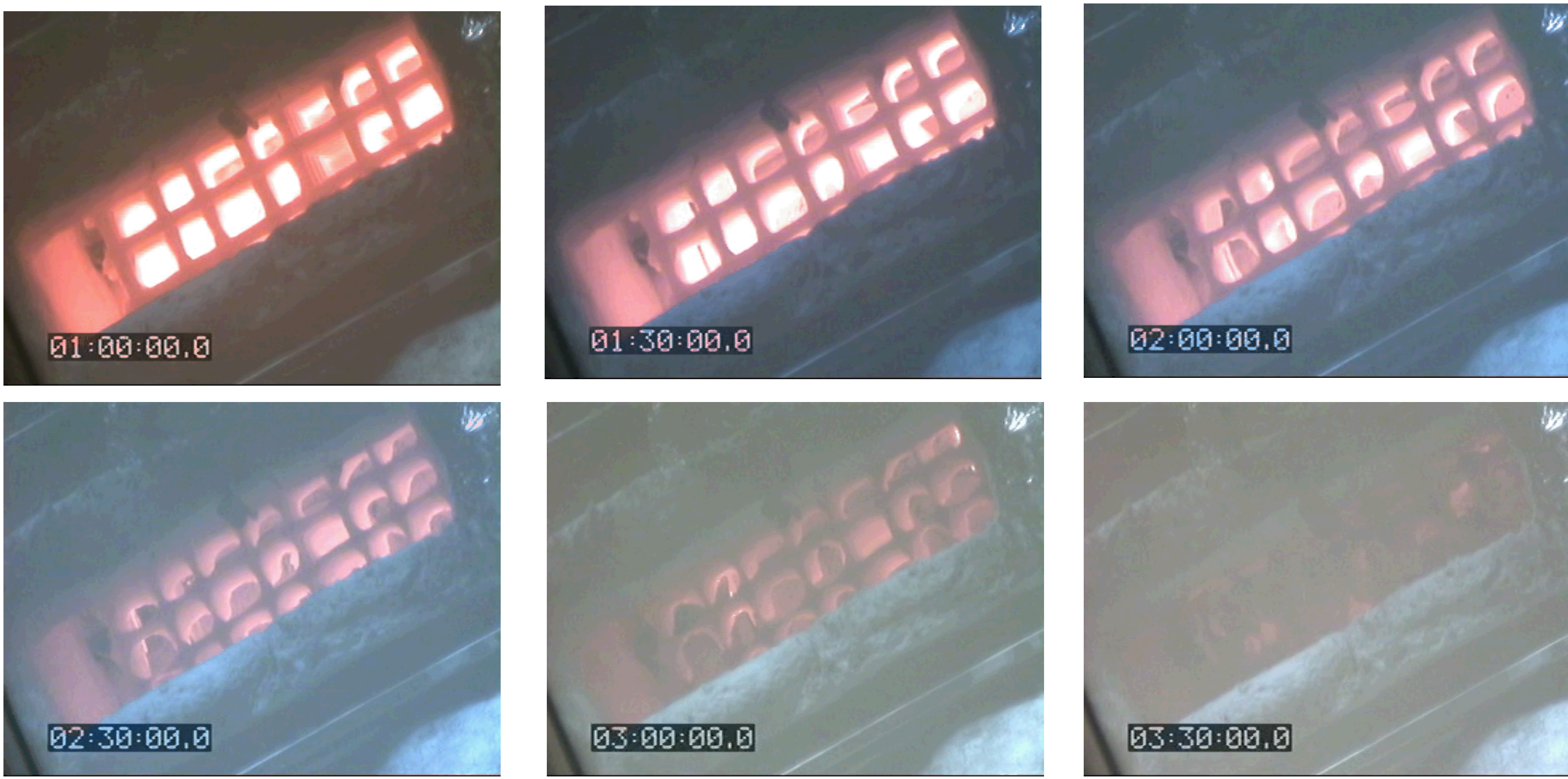
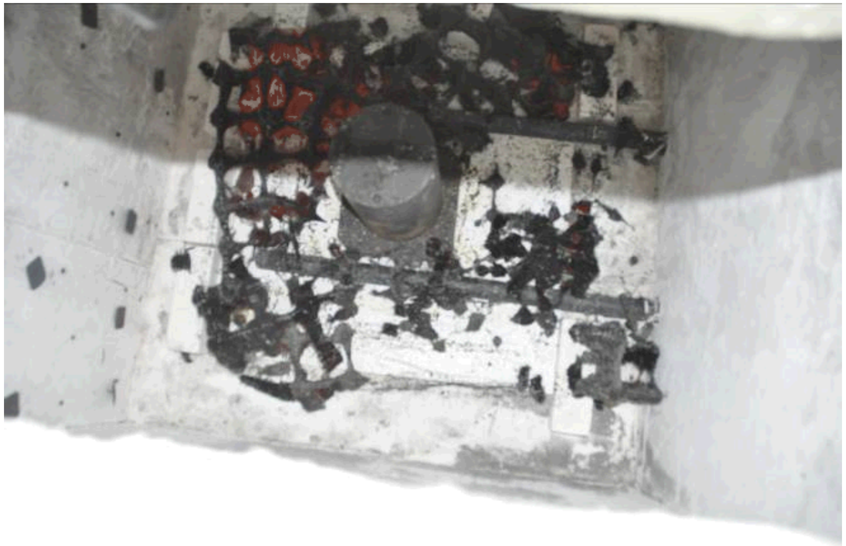
Test 6 (Strips) Video Frames



Frames show the progression of the reactions through the glowing combustion phase

Test 6 (Strips) Stills



Late-term burnout pattern evident in still photography
Air inlet is from the top in these photographs
Glowing nearest the air inlet at late burn-out.
Final mass: 6.7% of the original mass

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Test 6 (Composite Strips) Video

Testing Summary

Table 6. A summary of various results from six tests.

	Test	Initial Mass	Residual Mass	Peak Flux	Flaming Duration	Total Duration	SA/V	Mean Consumption Rate
	#	*kg*	*%*	*kW/m²*	*min*	*min*	*cm⁻¹*	*g/s*
Wood	1	40.8	-	220	-	90	2.4	7.56
	2	31.8	-	220	-	60	1.3	8.82
Composites	4	36.5	9.56	180	25	330	-	1.84
	5	38.5	2.59	175	30	420	2.0	1.53
	6	39.3	6.74	220	20	300	9.2	2.18
	7	26.5	10.34	160	10	240	6.9	1.84

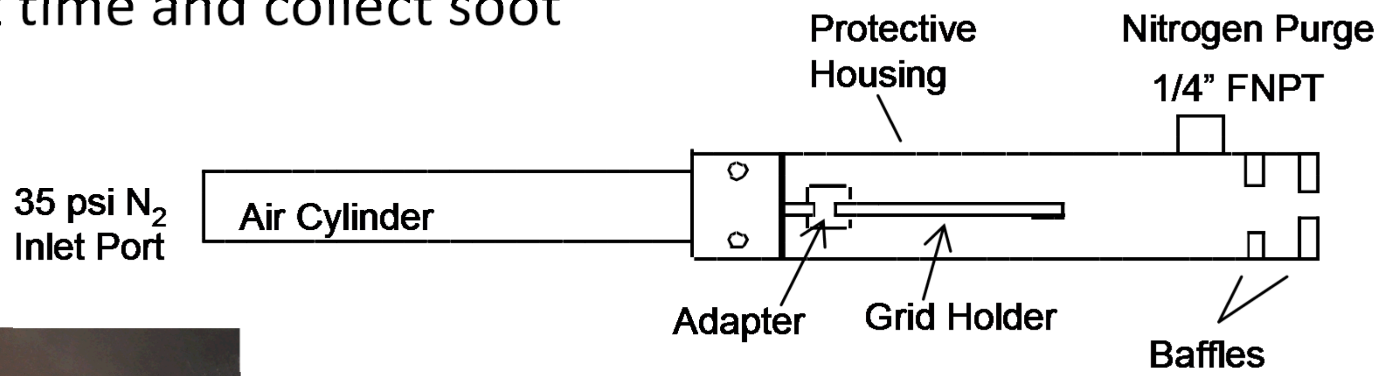
Compared to wood, peak fluxes tend lower, consumption rates are much lower, thermal release duration is much longer.

Surface Area to Volume appears to relate to consumption rate.

Very low residual mass; conflicts with observations from events.

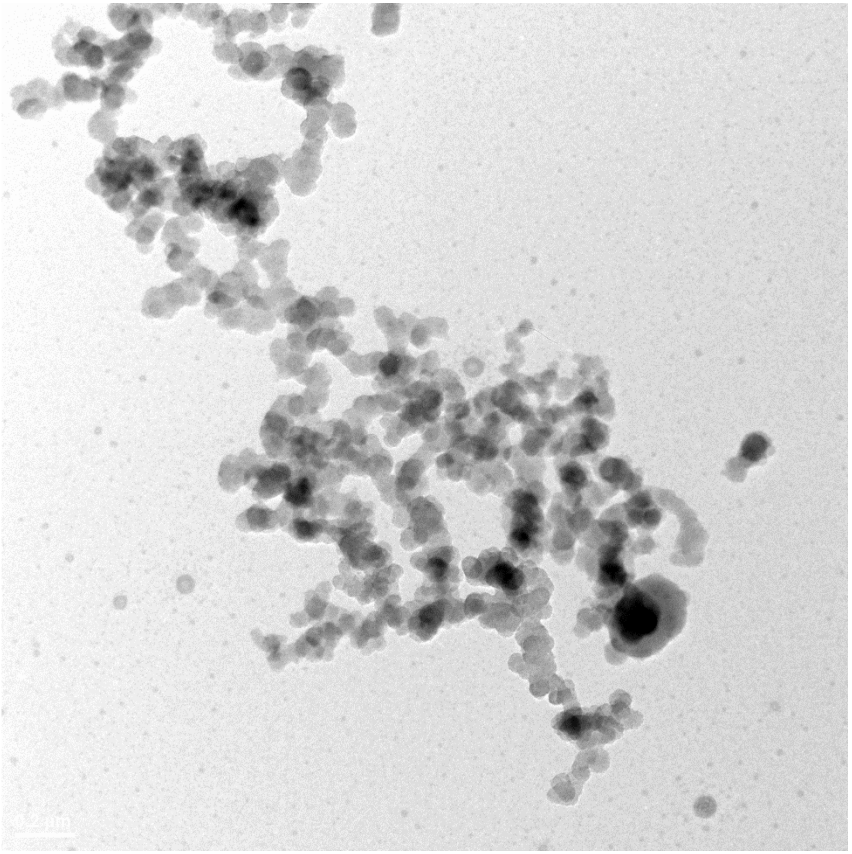
Soot Sampling System

Pneumatically actuated arm used to expose grid sources to the fire for a short time and collect soot

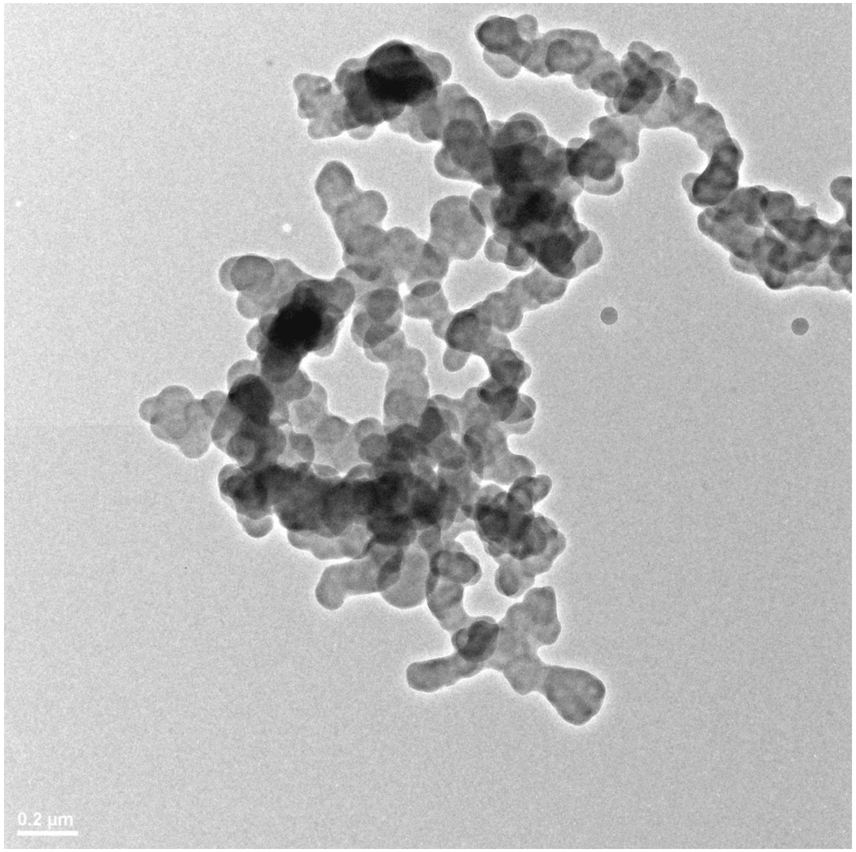


TEM Images of Soot

Test 5



Test 6



Larger soot spheres for Test 6 (same scale)

Both show large agglomerates

Test Materials

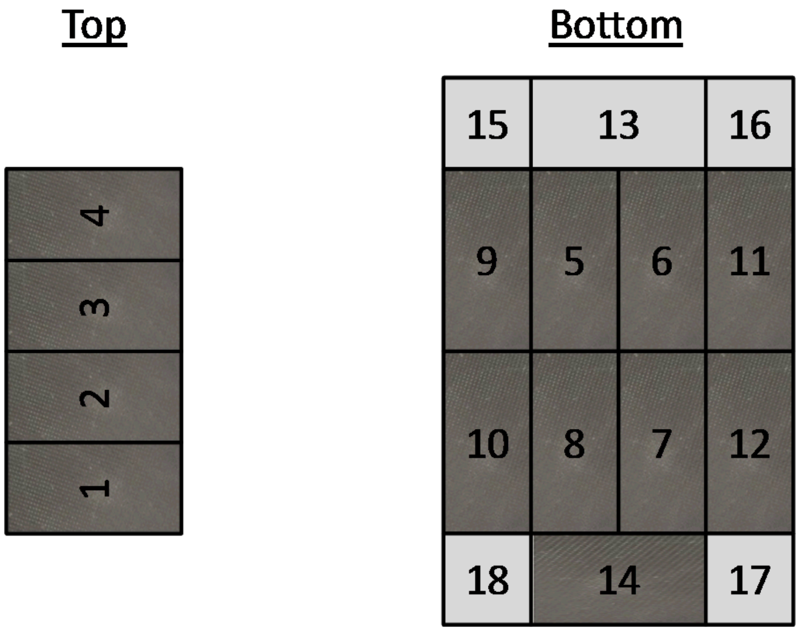
- 22 4' x 8' panels
  - 12 of which were sandwich panels with aluminum or paper core
  - Remainder were flat panels
- Simulation Matrix:

Photograph of a single panel

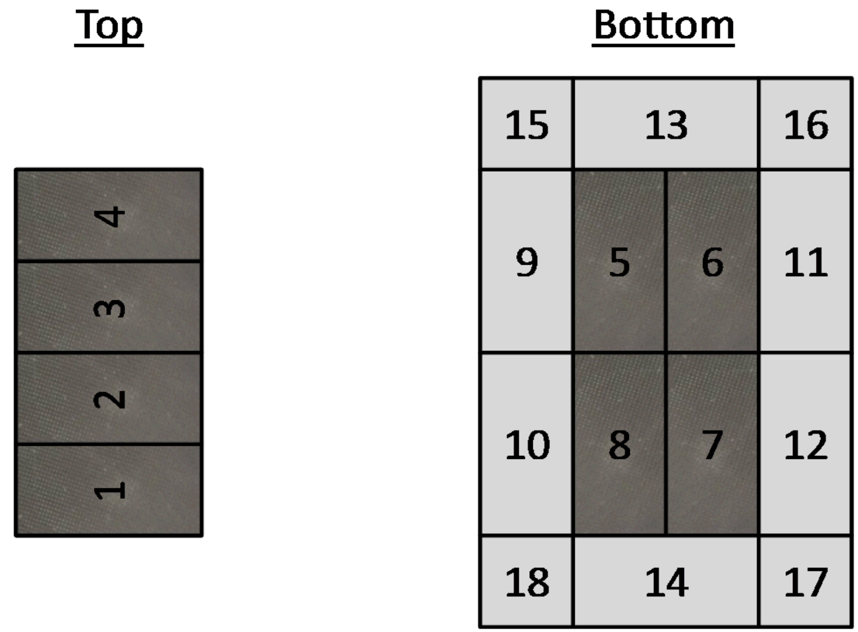
Series	Test #	Accelerant	Composite Mass (kg)	Air Speed (mph)	Epoxy Material	Reference
Enclosure	5	Propane	38.5	Variable	Hercules 3501-6	[9,15]
Enclosure	6	Propane	39.4	Variable	Hexcel 3501-6	[9,15]
Mock Aircraft	A	JP-8	206	8	ACG/Umeco MTM45-1 Hercules 8551-7A	[16]
Mock Aircraft	B	JP-8	179	5	ACG/Umeco MTM45-1 Hercules 8551-7A	[16]

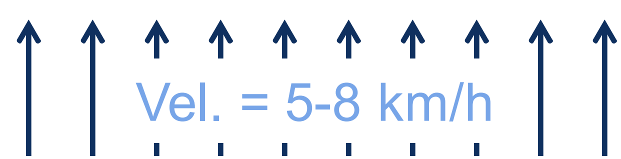
Panel Layout

High-wind scenario



Low-wind scenario





Fuel pan was located
under Panel 14, 7, 8

Photographs from Test A (high-wind)

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Photographs from Test B (low-wind)

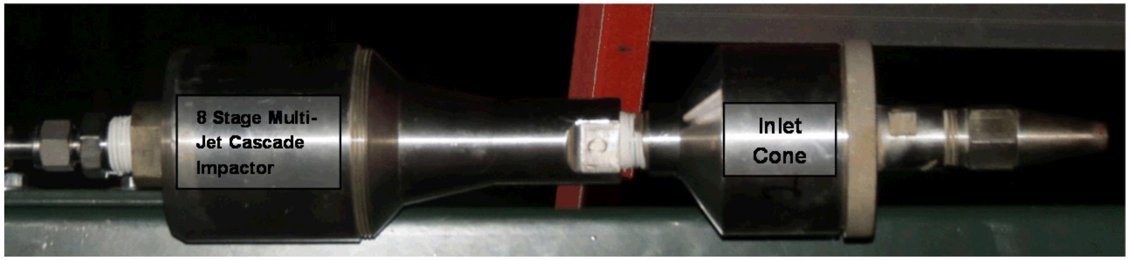
Photographs from Test B (low-wind)

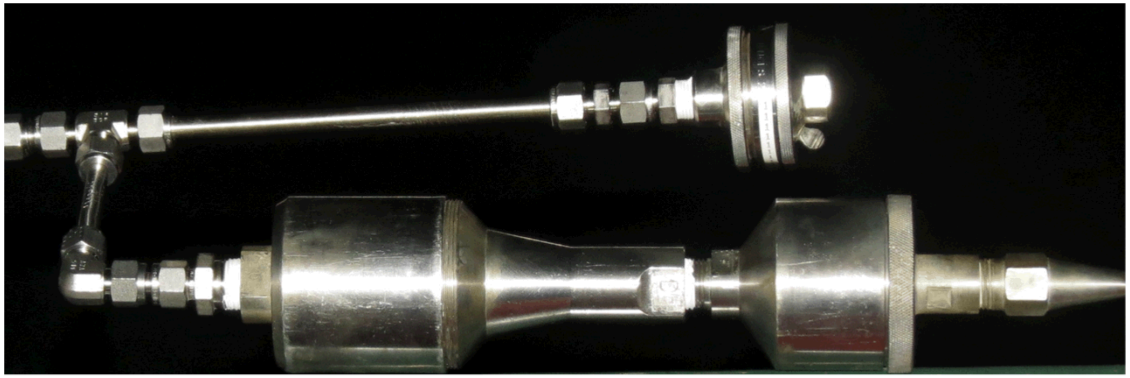
Photographs from Test B (low-wind)

Photographs from Test B (low-wind)

Cascade Impactor

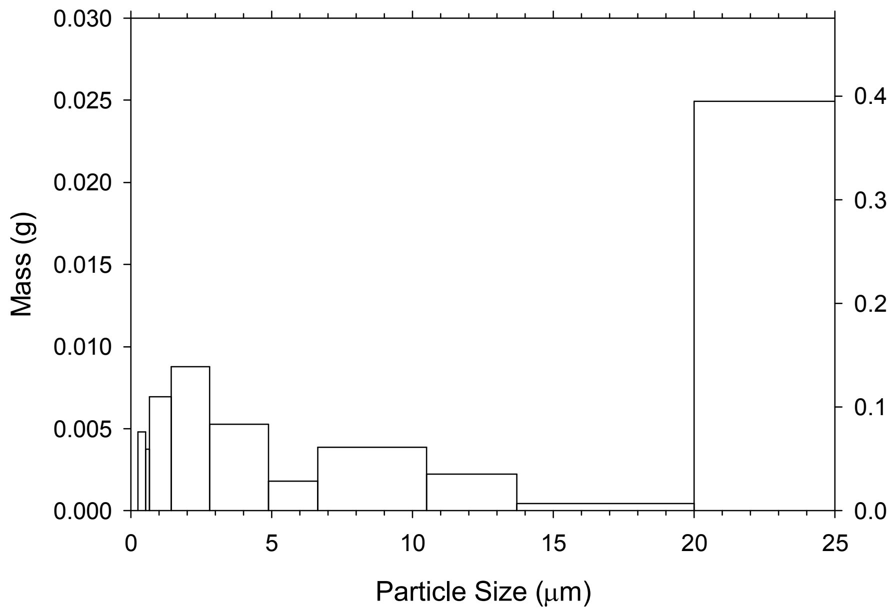
- Two impactors were located down-stream of the fire
  - 6 feet and 12 feet high on the same pole
- Sampling was active until a pre-defined pressure drop was achieved
- Impactors aerodynamically separate particles by size

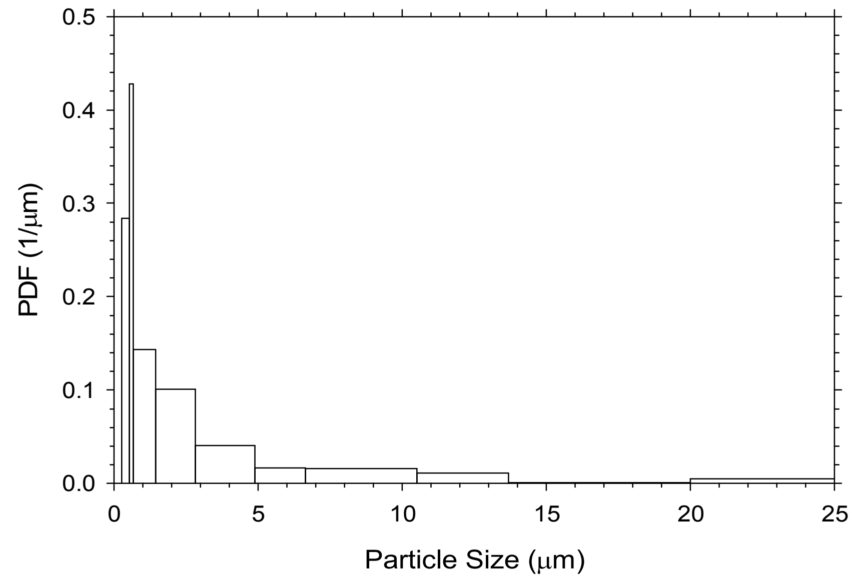




Mass and PDF of Particles from Impactor

- Impactor particle sampling suggests particles are most likely in the 0.1-1 μm range
- The distribution PDF appears smooth and regular
- These are thought to be primarily soot particles, although they may contain particles from other sources





Fiber and Soot Sampling

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Test	Measured Fibers [Fibers/cm^3]	Occupational Exposure Limit [Fibers/ cm^3]
A	0.07967	1.0
B	0.07502	1.0

- In both tests, the measured fibers were significantly below the occupational exposure limit
- Similarly for the carbon black sampling:
  - 0.478 mg/m^3 carbon black
  - Regulatory limit of 3.5 mg/m^3
- Sampling active during clean-up

Discussion

- Clean-up procedures initially involved vacuuming and wet wiping of surfaces
- Subsequent exposures were significantly below hazard thresholds:
  - This, even though material quantities were high (> 500 lbs)
  - This result was moderately surprising given the historical issues documented in the literature on carbon fiber epoxy materials
- Initial procedures may have contributed to this finding
- Even though our clean-up would have been safe without respirators, we are not recommending a change in procedure:
  - Real aviation crashes have more material, more material types
  - Clean-up was the day following the test, allowing material to settle or dissipate

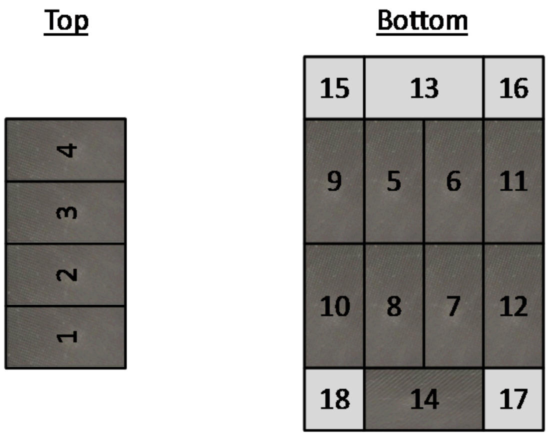
Summary and Conclusions

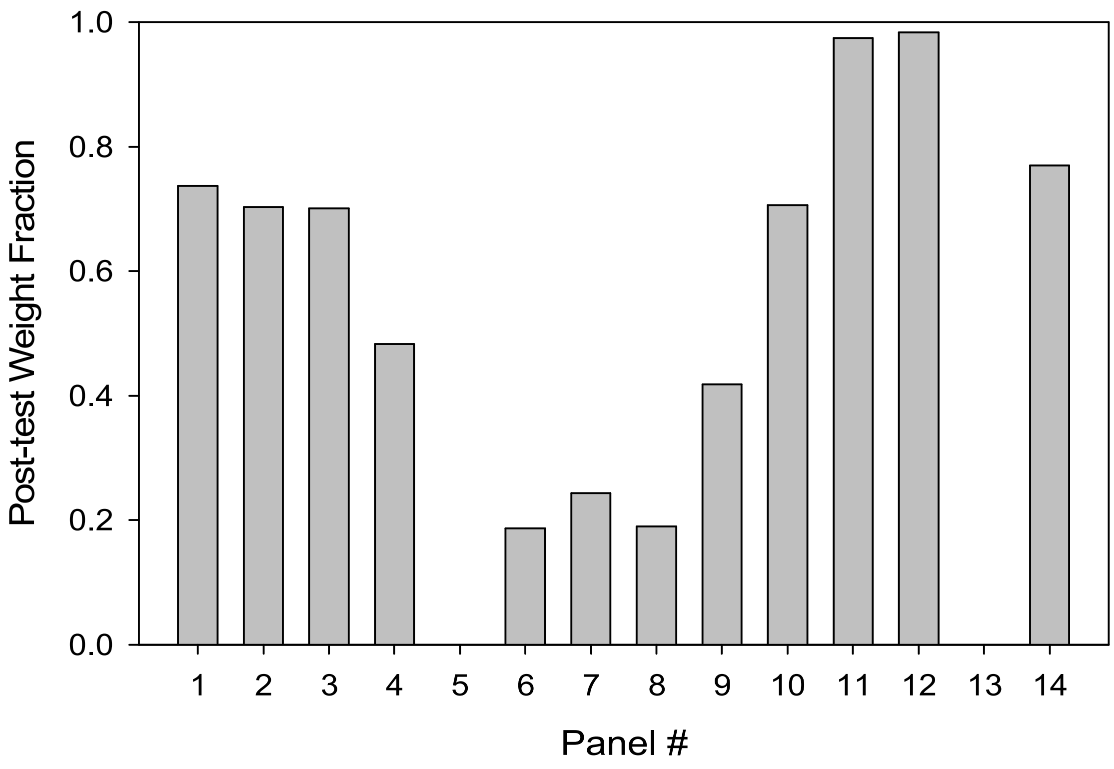
- Soot from the epoxy burning appears much like soot from liquid hydrocarbon fuel under TEM imaging.
- A cascade impactor extracted soot particle distributions, suggesting the most likely particles are found in the 0.1-1 μm range.
- Fiber and carbon sampling taken from a member of the clean-up crew suggested that the post- test hazard was not high enough to exceed standard exposure thresholds.
- These results add to the existing body of work on the health hazards of carbon fiber epoxy materials when they are a significant component of a fire.

Extra Viewgraphs

Post-test weight for Test A (high-wind)

- Panel 5 was not recovered
- Panels 11 and 12 were shielded by panel 6 and 7, did not burn
- Approximately 50% of initial mass recovered





Typical Test A heat flux measurements

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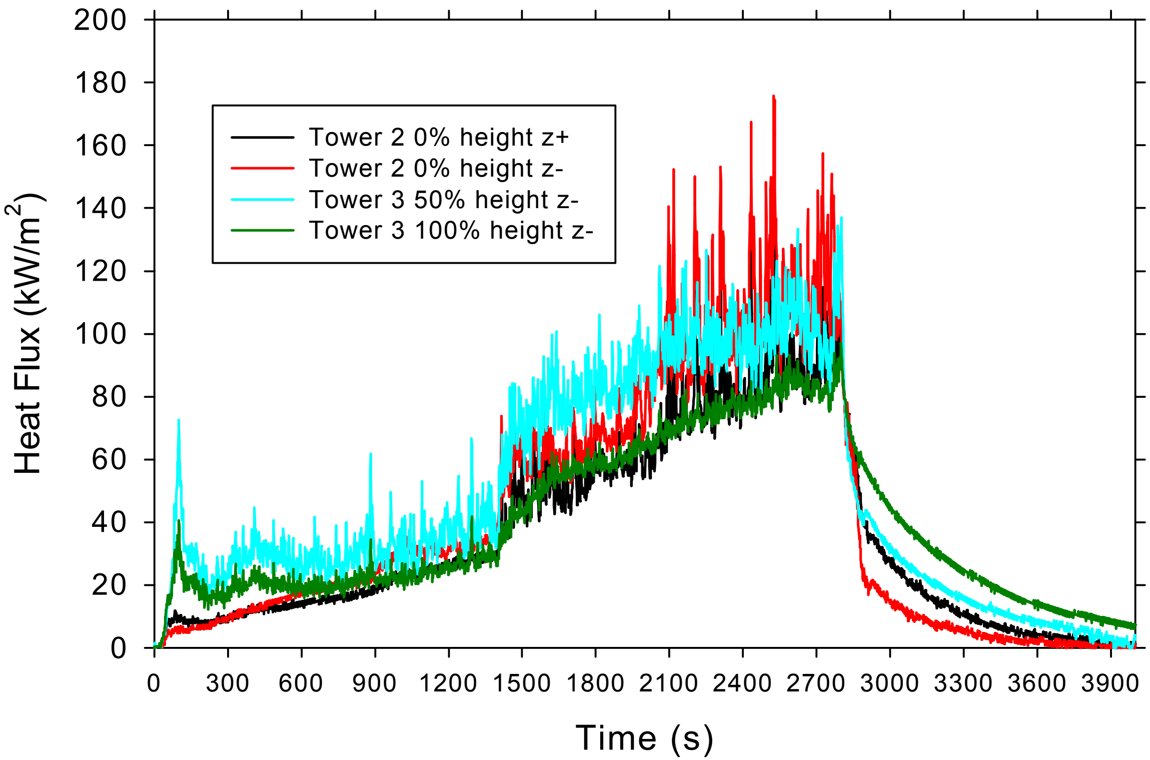
“Towers” had multiple heat flux gauges to measure temperature and heat flux



JP-8 fuel fire in circular pan under mock bay; ~45 minute burn typical of liquid fire durations

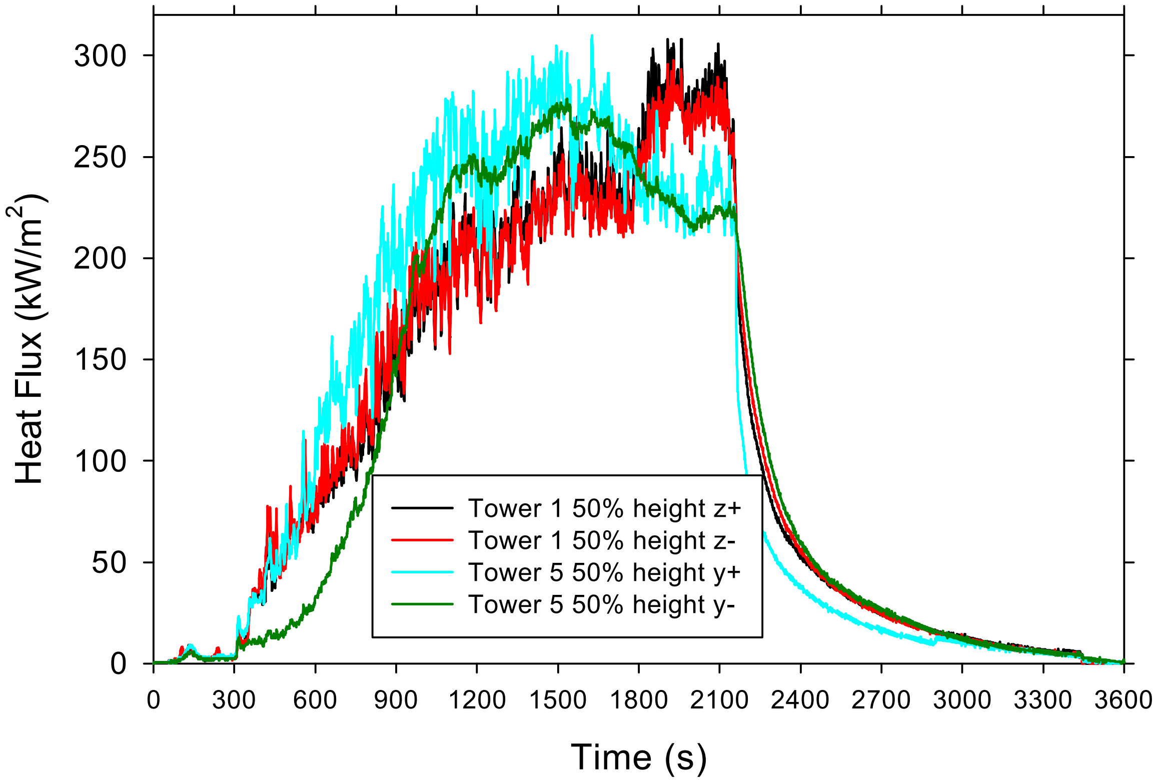
Slow, steady rise in flux to ~ 140kW/m^2

Inflection at 1500 second due to panel falling

Typical Test A heat flux measurements

"Towers" had multiple heat flux gauges to measure temperature and heat flux

JP-8 fuel fire in circular pan under mock bay; ~35 minute burn typical of liquid fire durations



Big difference; much faster rise, much higher flux (~320 kW/m^2)
Inflection at 300 seconds due to first panel falling

Why were fluxes so different?

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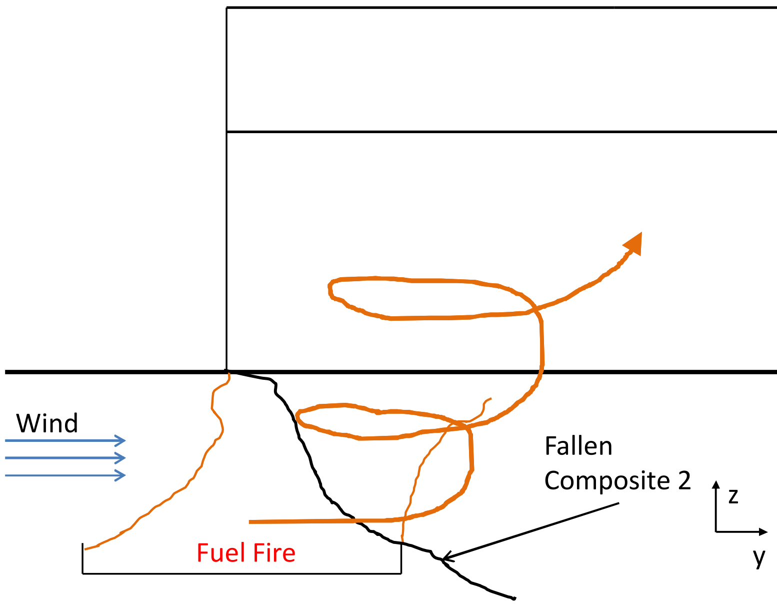
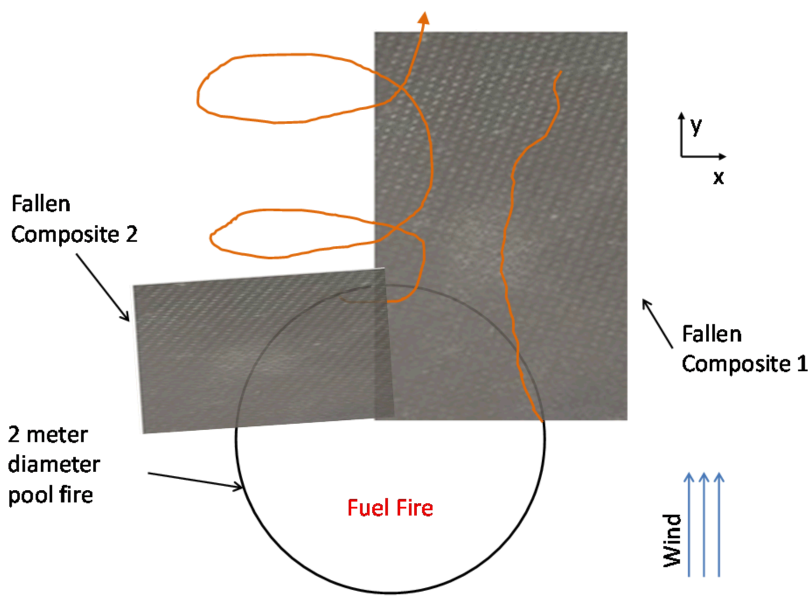
Vortical flows generated around a fallen composite panel
High mixing and burn rate caused by fuel/flow interaction

Side View



Top View

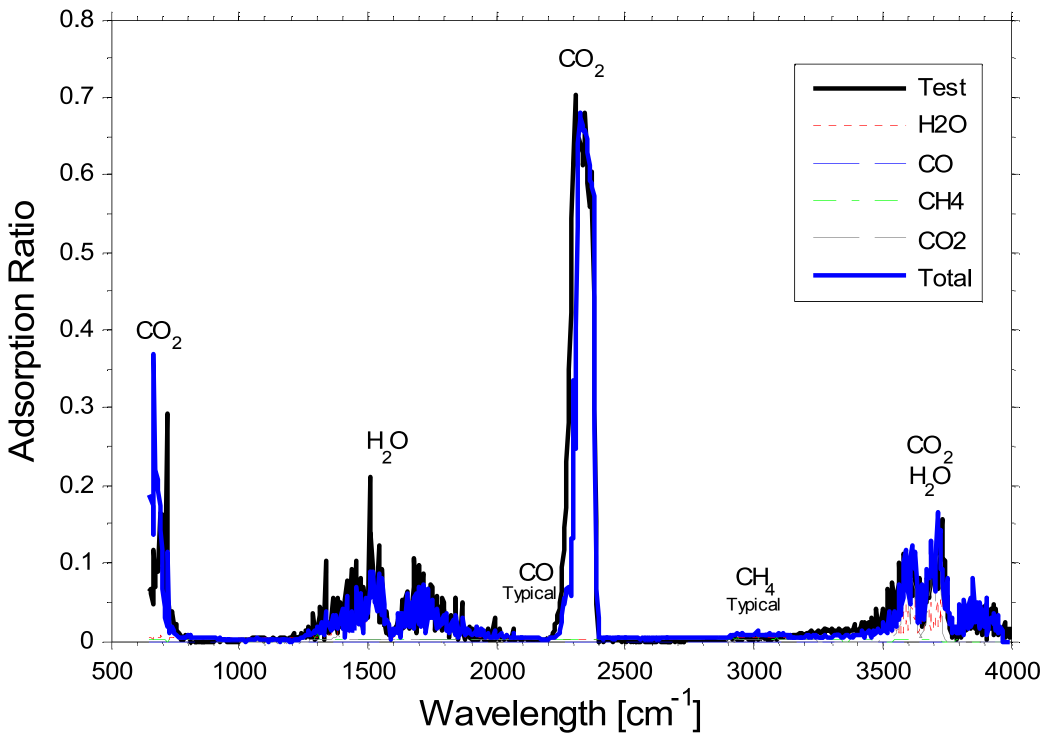


Test Setup and Pre-test

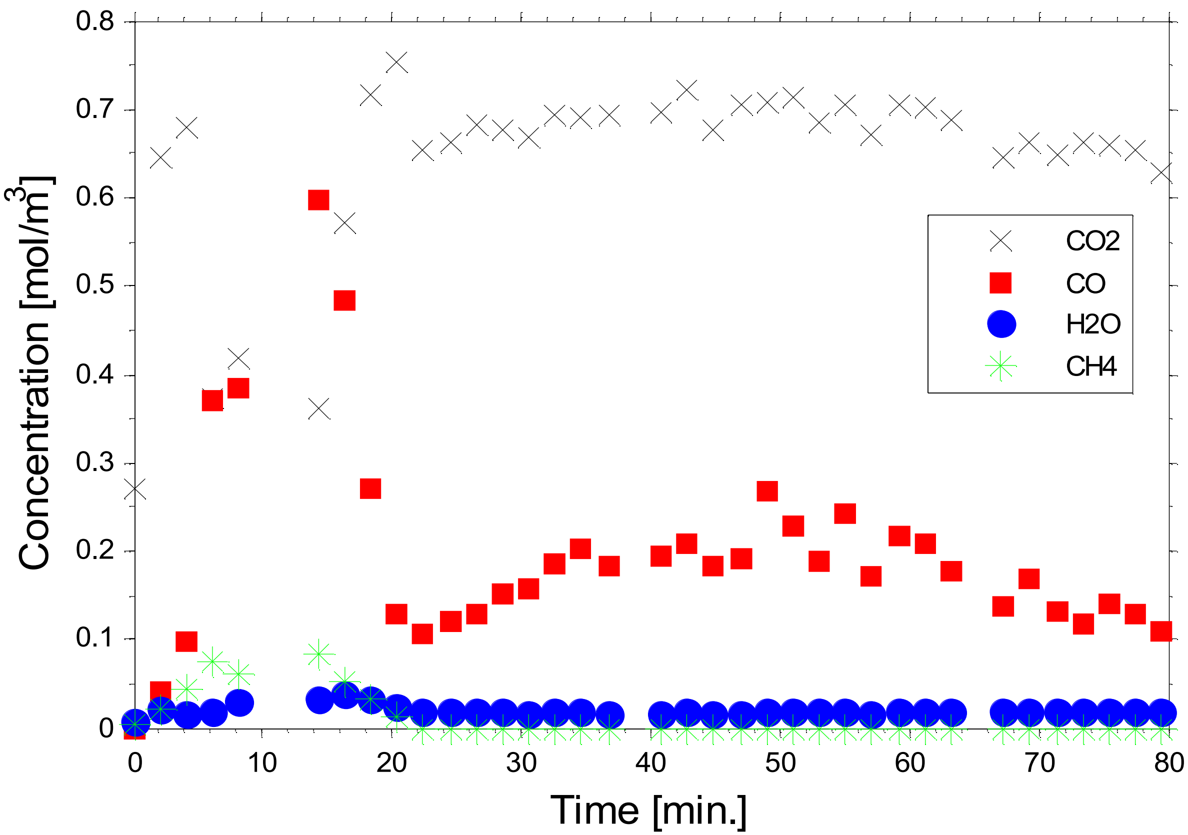
Test Setup and Pre-test

FTIR data from Enclosure Tests

Typical extraction



Test 5 (Body Armor) FTIR Results



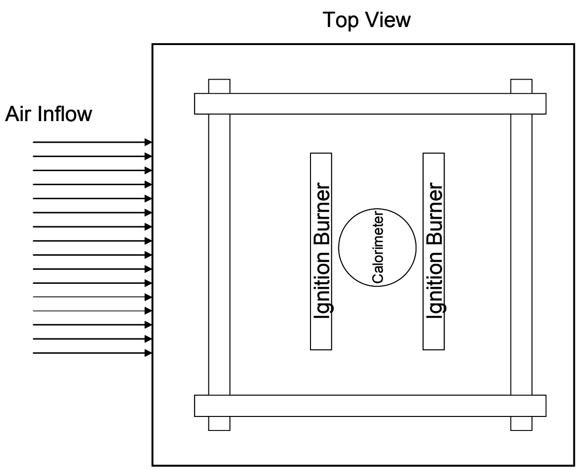
Low signal during peak flaming

H2O, CO, and CH4 peak during flaming, CO persistent during glowing combustion

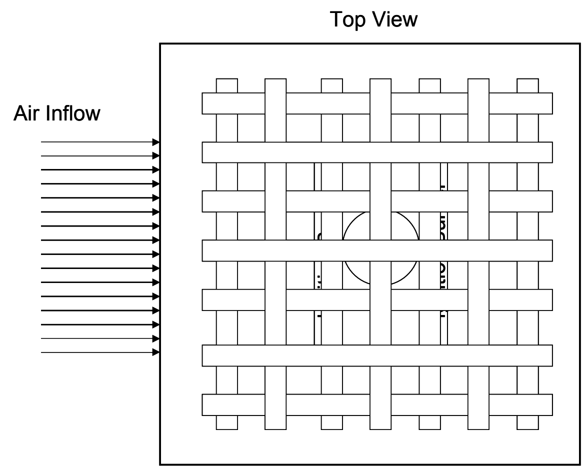
Test Setup and Fire Pics

Test 7 Layout

Lower Layer



Upper Layer



→ Air inflow was significantly varied for this test

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Test 7 (I-beams) Video Frames

Frames show the progression of the reactions through the glowing combustion phase

Test 7 (I-beams) Video